Evidence of a Non-Cubic Phase Transition During the Dehydration of Zinc-Exchanged Zeolite A.

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Introduction: In a recent study of zinc-loaded zinc-exchanged zeolite A it was found that zinc-oxide clusters were formed instead of zinc clusters [1]. It was evident that the non-framework oxygen in this sample came from residual water or hydroxide left over from the dehydration process. In an attempt to find a temperature at which Zn-A would be completely dehydrated, diffraction studies were undertaken. It was hoped that structural models could be obtained at various temperatures to monitor the amount of non-framework oxygen in the zeolite.

Methods and Materials: Zinc-exchanged zeolite A was produced from Na-A by conventional aqueous ion-exchange methods. Small amounts of the sample were dehydrated at 200°C, 400°C, 600°C and 800°C under vacuum. For diffraction studies to be carried out the samples were then packed into 0.7mm glass capillaries under an inert atmosphere and sealed with epoxy resin. The wavelength used for the samples dehydrated at 200°C and 400°C was 0.700189Å, while 1.150184Å was used for the sample dehydrated at 400°C and 1.150085Å for the sample dehydrated at 800°C.

Results: Hydrated Zn-A at room temperature has cubic symmetry. The results here indicate that the sample dehydrated at 200°C is not cubic, but instead it is rhombohedral, whilst the sample dehydrated at 600°C can be indexed as cubic. Dehydration under vacuum at 800°C causes damage to the framework and a loss of crystallinity. Examination of the diffraction data obtained from the sample at 400°C suggests it is a mixture of the rhombohedral and cubic phases. Work is currently in progress to obtain detailed structural models at each of these temperatures.

Conclusions: The results so far show a rhombohedral phase transition during the dehydration process of Zn-A but that above 600°C Zn-A is cubic once again. A rhombohedral phase transition has previously been reported for dehydrated Na-A at low temperatures [2,3], however the work reported here is unusual as the phase transition occurs at elevated temperatures. As Zn-A reverts to cubic symmetry above 600°C it is logical to suggest that the rhombohedral phase transition may be caused by ordering of the non-framework atoms during initial dehydration.

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References: [1] J.E. Readman, I. Gameson, J.A. Hriljac, P.P. Edwards, P.A. Anderson, Chemical Communications, 595, 2000. [2] J.M. Bennett, C. S. Blackwell, D.E. Cox, ACS Symposium Series, **218**, 143, 1983. [3] L.A. Bursill, E. A. Lodge, J.M. Thomas, A.K. Cheetham, Journal of Physical Chemistry, **85**, 2409 1981.

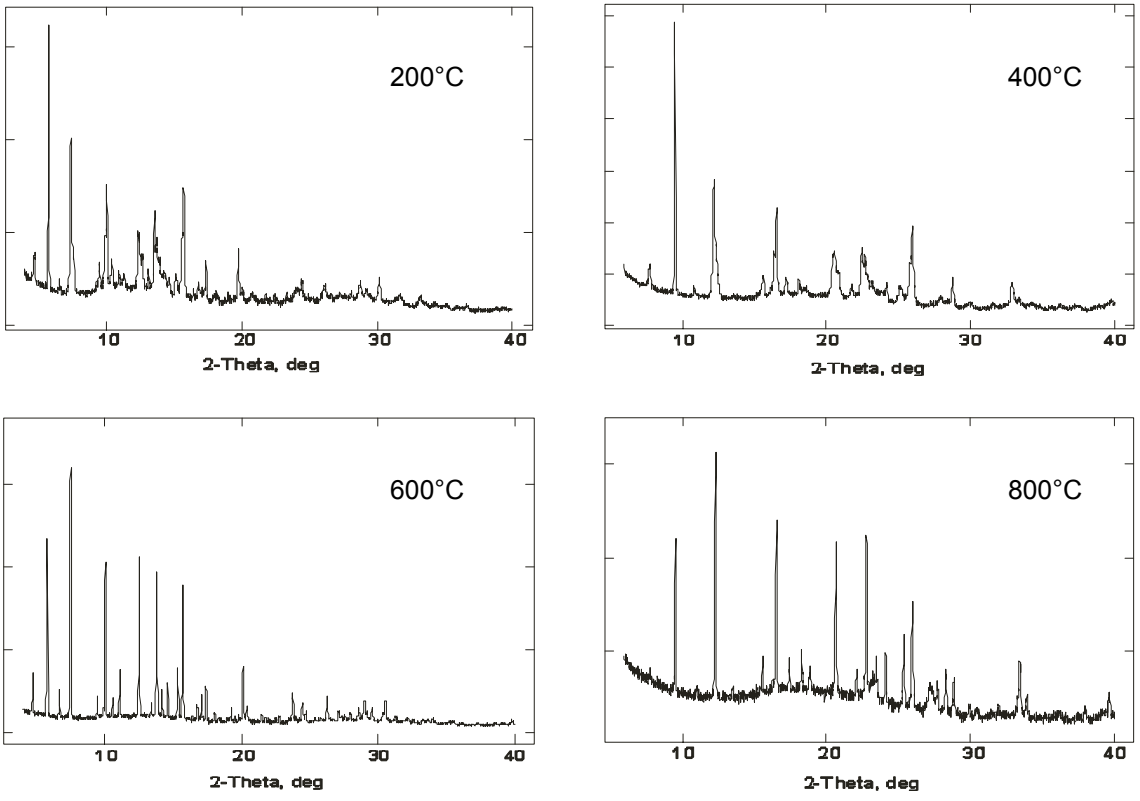

Figure 1: Diffraction patterns of Zn-A dehydrated at various temperatures.